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MEMORANDUM

DATE: 23 December 1998

TO: David Bennett, WAM, U.S. EPA, Region X

FROM: Michelle Turner, Chemist, WESTON, Seattle
Roger McGinnis, Senior Environmental Chemist, WESTON, Seattle

SUBJECT: Validation of Organotin Data
Laboratory Batch K9806374
Site. Duwamish River

WORK ASSIGNMENT NO. 46-35-0JZZ

WORK ORDER NO.: 4000-019-038-5200-00

DOC CONTROL NO.: 4000-019-038-AAAK

cc: Bruce Woods, RAP-WAM, U.S. EPA, Region X
Dena Hughes, Site Manager, WESTON, Seattle (memo only)
Kevin Mundell-Jackson, Database Management, WESTON

The quality assurance review of three sediment samples, laboratory batch K9806374, collected from the Duwamish River has been completed. The sediment samples were analyzed for organotins by Columbia Analytical Services of Kelso, Washington. Samples were analyzed by gas chromatography with an FPD detector. The samples were numbered:

98384002 98384003 98384008

Data Qualifications

The following comments refer to the laboratory performance in meeting the quality control criteria described in the technical specifications of the laboratory subcontract. The review follows the format described in the *National Functional Guidelines for Organic Data Review* (EPA OSWER Directive 9240.1, February 1994), modified to include specific requirements of analytical methods.

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1. Timeliness

All samples were extracted 43 days after sample collection, exceeding the 7 day holding time criteria in the Sampling and Analysis Plan. However, prior to extraction, samples were stored frozen, thus extending the holding time. Samples were extracted within the 12 month holding time recommended by PSEP for frozen samples.

2. Detection Limits

Detection limits met project required quantitation limits with the following exceptions:

Sample	Compound	QL Goal (µg/Kg)	Reported QL (µg/Kg)
98384002	Tetrabutyltin	10	15
98384003	Tetrabutyltin	10	15
98384008	Tetrabutyltin	10	15

Where quantitation limit goals were exceeded, undetected analytes were qualified (UI) to indicate matrix interference.

3. Initial Calibration

A seven-point initial calibration was performed prior to each analytical batch. The percent relative standard deviation for the initial calibration was within limits of less than 25 percent RSD.

4. Continuing Calibrations

Continuing calibration check was performed after every 10 samples. All target analytes were within required limits for the continuing calibrations with the percent difference for a mid-range standard less than 25 percent.

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5. Blanks

a) Laboratory Method Blanks

Laboratory method blank frequency criteria were met. No target analytes were reported in laboratory method blanks

b) Field Blanks

No field blanks were associated with this SDG.

6. Surrogate Compound Recovery

Surrogate recovery goals for Tripropyltin were established in the project Sampling and Analysis Plan at 60 to 120 percent for sediment. Based on conversations with the laboratory an additional surrogate, Triphenyltin was added and historical laboratory control chart limits were also used for data qualification. Laboratory limits are presented below:

Surrogate Compound	Sediment Limits
Tripropyltin	18 - 125%
Triphenyltin	28 - 122%

Surrogate compound percent recoveries were outside the QC limits for the following samples

Sample	Surrogate	Percent Recovery
98384003	Tripropyltin	45
98384003	Triphenyltin	46

Samples results and detection limits were qualified as estimated (UJ/J) when both surrogate recoveries were outside the QC limits.

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7. Laboratory Control Sample (LCS)

LCS recovery goals for Butyltins were established in the project Sampling and Analysis Plan at 60 to 130% for sediment. Based on conversations with the laboratory, historical control chart limits of 8 to 161 percent for sediment were also used for data qualification.

Laboratory control sample percent recoveries met QC guidelines (P-project, L-laboratory), with the following exceptions:

LCS	Analyte	Percent Recovery	QC Limit	Associated Samples
K981027-LCS	Dibutyltin	45	60-130 (P) 8-161 (L)	98384002 98384003 98384008
K981027-LCS	n-Butyltin	18	60-130 (P) 8-161 (L)	98384002 98384003 98384008

Sample results were qualified as estimated (J) when LCS recoveries were outside project limits. Undetected results were also qualified as estimated (UJ) when LCS recoveries were outside project limits.

8. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

The following matrix spike recovery goals were established in the project Sampling and Analysis Plan at for sediment

Analyte	% Recovery
Tributyltin	40 - 120%
Dibutyltin	30 - 120%
n-Butyltin	10 - 120%

MS/MSD sample percent recoveries met QC guidelines (P-project, L-laboratory), with the exception of the following:



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Sample	Compound	Percent Recovery	QC Limits
K9806404-007MS (Batch QC)	Dibutyltin	16	30-120 (P) 8-144 (L)
K9806404-007DMS (Batch QC)	Dibutyltin	20	30-120 (P) 8-144 (L)

MS and MSD recoveries for n-Butyltin were not calculated due to matrix interferences.

Relative percent differences (RPD) between the MS and MSD percent recoveries met QC guidelines for compounds. No action was based solely on MS/MSD data.

9. Field Duplicate Analysis

No field duplicates were associated with this SDG.

10 Sample Analysis

A cursory review of raw data was performed. Deliverables were accurate and complete. The case narrative indicated that the MS recovery of n-Butyltin in the Batch QC sample was outside the QC limits because of suspected matrix interference. Also, the LCS was reanalyzed because n-Butyltin results in the initial analysis were outside the laboratory QC limits. The n-Butyltin results from the reanalysis on 11/4/98 were reported with the laboratory QC results. No other problems were noted in the case narrative.

11 Laboratory Contact

No laboratory contact was required.

Data Assessment

Upon consideration of the data qualifications noted above, the data are ACCEPTABLE for use except where flagged with data qualifiers that modify the usefulness of the individual values.

Data Qualifiers

U - The compound was analyzed for, but was not detected.

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- UJ - The compound was analyzed for, but was not detected. The associated quantitation limit is an estimate because quality control criteria were not met
- J - The analyte was positively identified, but the associated numerical value is an estimated quantity because quality control criteria were not met or because concentrations reported are less than the quantitation limit or lowest calibration standard
- R - Quality control indicates that data are unusable (compound may or may not be present). Resampling and reanalysis are necessary for verification.
- N - Presumptive evidence of presence of material (tentative identification).
- I - Elevated reporting limit due to matrix interference.

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COLUMBIA ANALYTICAL SERVICES, INC.

Analytical Report

Client: Roy F Weston, Inc
Project: Duwamish River/4000-027-001-2019-38
Sample Matrix: Sediment

Service Request: K9806374
Date Collected: 9/14/98
Date Received: 9/15/98

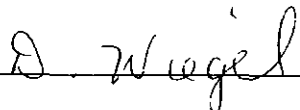
Butyltins

Sample Name 98384002 Units: ug/Kg (ppb)
Lab Code K9806374-003 Basis: Dry
Test Notes D

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	15	5	10/27/98	10/31/98	ND	UI
Tri-n-butyltin	Method	Butyltins	5	5	10/27/98	10/31/98	216	
Di-n-butyltin	Method	Butyltins	5	5	10/27/98	10/31/98	82	J
n-Butyltin	Method	Butyltins	5	5	10/27/98	10/31/98	66	J

D The MRL is elevated because of matrix interferences and because the sample required diluting

Approved By



Date

11/11/98

1S22/020597p

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Analytical Report

Service Request: K9806374
Date Collected: 9/14/98
Date Received: 9/15/98

Units. ug/Kg (ppb)
Basis Dry

Analyte	Prep	Analysis	MRL	Dilution	Date	Date	Result	Result
	Method	Method		Factor	Extracted	Analyzed		Notes
Tetra-n-butyltin	Method	Butyltins	15	5	10/27/98	10/31/98	ND	UIJ
Tri-n-butyltin	Method	Butyltins	5	5	10/27/98	10/31/98	31	J
Di-n-butyltin	Method	Butyltins	5	5	10/27/98	10/31/98	9	J
n-Butyltin	Method	Butyltins	5	5	10/27/98	10/31/98	ND	uJ

11/12/15/18

Date _____

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COLUMBIA ANALYTICAL SERVICES, INC.

Analytical Report

Client: Roy F Weston, Inc
Project: Duwamish River/4000-027-001-2019-38
Sample Matrix: Sediment

Service Request: K9806374
Date Collected: 9/14/98
Date Received: 9/15/98

Butyltins

Sample Name 98384008
Lab Code K9806374-009
Test Notes D

Units: ug/Kg (ppb)
Basis Dry

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
Tetra-n-butyltin	Method	Butyltins	15	5	10/27/98	10/31/98	ND	UI
Tri-n-butyltin	Method	Butyltins	5	5	10/27/98	10/31/98	92	
Di-n-butyltin	Method	Butyltins	5	5	10/27/98	10/31/98	24	J
n-Butyltin	Method	Butyltins	5	5	10/27/98	10/31/98	11	J

D The MRL is elevated because of matrix interferences and because the sample required diluting

Approved By D. Weigel Date 11/11/98

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20972/15/98